

INTELLIGENCE

50X1-HUM

CENTRAL INTELLIGENCE AGENCY

INFORMATION REPORT

CONFIDENTIAL

50X1-HUM

COUNTRY	USSR (Voroshilovgrad Oblast)	REPORT	
SUBJECT	Research in Dyes and Dye Intermediates at NIOPIK, Rubezhnoye	DATE DISTR.	8 January 1954
		NO. OF PAGES	9 50X1-HUM
DATE OF INFO.		REQUIREMENT	
PLACE ACQUIRED		REFERENCES	

50X1-HUM

THE SOURCE EVALUATIONS IN THIS REPORT ARE DEFINITIVE.
THE APPRAISAL OF CONTENT IS TENTATIVE.
(FOR KEY SEE REVERSE)

50X1-HUM

CONFIDENTIAL

STATE	#x	ARMY	#x	NAVY	#x	AIR	#x	FBI		AEC		OSI	Ev	x		
-------	----	------	----	------	----	-----	----	-----	--	-----	--	-----	----	---	--	--

(Note: Washington Distribution Indicated By "X"; Field Distribution By "#")

50X1-HUM

50X1-HUM

C O N F I D E N T I A L

REPORT

COUNTRY : USSR (Voroshilovgrad Oblast)

DATE DISTR. 27 NOV 53

SUBJECT : Research in Dyes and Dye Intermediates at
NIOPiK, Rubezhnoye

NO. OF PAGES 8

PLACE
ACQUIRED :NO. OF ENCLS.
(LISTED BELOW)

50X1-HUM

DATE
ACQUIREDSUPPLEMENT TO
REPORT NO.

DATE OF INFORMATION :

THIS IS UNEVALUATED INFORMATION

50X1-HUM

RESEARCH ON DYES FOR COLOR PHOTOGRAPHY AT RUBEZHNOYE

1. In 1946, a group of 23 German organic and physical chemists [redacted] were transported to a development laboratory at Rubezhnoye, USSR, to work on development problems connected with the manufacture of known German dyes by the factory "Chimkombinat" at Rubezhnoye. Filiale NIOPiK was a laboratory in Rubezhnoye specifically organized by NIOPiK (Nauchnyi Institut Organicheskoy Promyshlennosti-Produktov e Krasiteleyi), Moscow, in order to permit the transported German group to work within the USSR under Soviet direction, but at the same time to restrict their activities to their own laboratory. Liaison between the Moscow NIOPiK office and the laboratory, and between both the "Chimkombinat" plant and its technical service laboratory, was conducted by a single liaison officer. General problems were assigned to the group by the NIOPiK liaison officer, and were concerned with applied research only: i.e., adaptation of German processes for the manufacture of textile and color-photography dyes to Soviet manufacturing facilities.

50X1-HUM

50X1-HUM

C O N F I D E N T I A L

C O N F I D E N T I A L

- 2 -

ties and to available Soviet chemical intermediates.

50X1-HUM

2. [] German chemists who were selected by the Soviets primarily to work on the development of dyes and dye components for color photography [] were transported to Rubezhnoye in November 1946, and for a period of about nine months [] given laboratory space in the very poorly equipped and limited Central Laboratory of Chimkombinat. For some time a rumor had circulated that [] was eventually to be transferred to a new laboratory in Moscow that was ostensibly to be for the sole use of the technical group, but nothing further was heard, and instead [] moved in August 1947 to the new NIOPIK Branch Laboratory (Nauchnyi Institut Organicheskoy Promyshlennosti i Produktov e Krasiteleyi; Scientific Institute for Organic Dyes and Dye Intermediates) in Rubezhnoye. 50X1-HUM
3. Shortly after [] arrival in Rubezhnoye [] were visited by a committee of three that consisted of UVAROV, who was the representative of the Chemical Ministry that controlled NIOPIK; Prof. VOROSHOV, the scientific consultant to NIOPIK, Moscow; and Mrs. RASCHESKAYA, the head of the Central Laboratory of Chimkombinat. The latter interviewed each specialist and assigned him a problem. Later scientific One-Year Plans that included suggestions [] made were drafted. 50X1-HUM

Laboratory Poorly Equipped

4. The laboratory had its own library and in addition also had access to the fairly good Chimkombinat library, equipped with scientific literature in the form of journals, books, and both German and Soviet reports. Upon request [] could obtain documents from NIOPIK, Moscow. The laboratory was very poorly equipped, however, and particularly at the beginning lacked common supplies such as thermometers, flasks, condensers, etc. More complex items such as microscopes, refractometers, viscosimeters, constant temperature baths etc., could be obtained from the so-called "trophy storage," of German equipment. However, the needs of Central Laboratory took precedence over those of the NIOPIK Branch Laboratory. Balances were of Soviet manufacture, but were insensitive and of low precision according to German standards. As time progressed, the marked deficiency in laboratory equipment was alleviated by imports from Germany; and it was of some interest [] that [] better equipped than were equivalent laboratories in Germany with ground-glass articles due to these importations. Glassware of Soviet origin was available, but was of very poor quality; and it was only because of [] very gifted Soviet glassblower [] able to maintain adequate stocks of glass apparatus. 50X1-HUM
5. However, in no case can it be said that [] laboratory was well equipped, either with glassware or with chemicals. Common reagents such as sodium sulfate, potassium chloride, potassium bromide, etc., were unobtainable. It was necessary to prepare these reagents from the appropriate acids and bases or from stocks of reagents that had been originally imported from Germany, as for example potassium bromide, which was prepared from a small supply of potassium bromate that happened to be in stock. Purification of reagents for analytical purposes was almost always necessary, such as for example the purification of hydrochloric acid by distillation. The comparatively low availability of Soviet 50X1-HUM

C O N F I D E N T I A L

C O N F I D E N T I A L

- 3 -

50X1-HUM

chemicals was not a question of their availability; the USSR Norm Catalog listed a wide selection, but was rather the result of the cumbersome transportation system. For example, flammable chemicals were shipped for short distances only by truck, and the convenient German practice of shipping such materials by rail was unknown. On the whole, the quality of Soviet chemicals when delivered was rather poor.

6. Necessary tools such as slide rules, magnifying glasses, glass cutters, typewriters (German), were in very short supply, and were not provided by the Soviets. Paper was also in exceedingly short supply for all purposes, and for a long period it was necessary to use paper sacks or wrapping paper for the required reports. [redacted] typewriter paper of rather poor quality was made available on a limited basis.

50X1-HUM

7. In spite of all these difficulties, the German group managed to conduct significant work. [redacted] learned quickly to improvise, just as do the Soviets who are masters of improvisation, and it was quite obvious [redacted] expected to use all [redacted] knowledge in every aspect of [redacted] activities there.

50X1-HUM

50X1-HUM

50X1-HUM

Soviet Personnel of Inferior Caliber

8. Soviet laboratory assistants, mostly women, were at our disposal, and usually came untrained [redacted] except for a limited knowledge of German. As might be expected, a few were gifted and eager to learn and very eager to rectify their obvious lack of practical laboratory training. All Soviet technical personnel were required to take courses regularly and to take an examination once a year, from the results of which they were classified according to position. A few came with limited formal training, but this was the exception rather than the rule.

50X1-HUM

50X1-HUM

9. [redacted] less success with the Soviet chemists who occasionally came to work [redacted]. Their training at that time was poor, particularly in laboratory experience, and only a few of the younger chemists were willing to further their own education by self-study in order that they might prosecute a problem independently. On the whole, the average Soviet chemist seemed to be afraid to pursue independently his own problems, in part it seems because of his fear to accept responsibility for his own decisions and in part because of a well-defined inferiority complex. He had the tendency to follow written instructions uncritically (i.e., "cook-book chemistry") and to use formulae rather than reasoning, and consequently often gave the impression that he was rather ungifted for his work. On the whole, the relationships between the Soviet and the German chemists in the laboratory were quite good, and were free of invidious nationalistic or racial comparisons, but it was obvious that these social relationships could not be continued into private life.

50X1-HUM

RESEARCH PROJECTS AT NIOPIK BRANCH LABORATORY

50X1-HUM

10. [redacted] the NIOPIK Branch Laboratory was occupied with a number of projects, [redacted] briefly describe.

C O N F I D E N T I A L

50X1-HUM

C O N F I D E N T I A L

- 4 -

50X1-HUM

- a. Vat Dyes, especially Thioindigo Compounds: This project was assigned from the start and continued until the group was returned to Germany in 1951. Dr. WUTKE (now group leader at Wolfen) as advisor and planner, and Prof. Dr. RIECHE, Dr. HOFFMAN and, [] were occupied in this group throughout. Dr. W. RICHTER and Dr. HALL were assigned occasional tasks. In 1949 the vat dye and anthraquinone compounds development section was expanded and Dr. GNUECHTEL, Dr. SCHULZE [] were assigned to the group. 50X1-HUM
- b. Dye Intermediates: At first, the projects in this class were those of interest to Chirkombinat. For example, Dr. CARO [] worked on the nitration of chlorobenzene and toluene and the isolation of the nitration products. Other problems included the preparation of intermediates such as trichlorobenzene (by Dr. ENGELMANN), 2, 6-dichlorotoluene (Dr. RICHTER), sulfonation of various naphthalenes (Dr. SCHULZE). In general, these syntheses were undertaken in order to produce small quantities of dyes as standards of purity. 50X1-HUM
- c. Dye Components for Color Photography: Several research assignments that had as their objective the improvement of quality of color-film dye components and improvements in the yields of the reactions that produced them were assigned to Dr. HALL and to Dr. GNUECHTEL.
- d. Indicators: In 1948 the Bayer series of indicators were produced in laboratory scale by Dr. THURM, Dr. SCHULZE, Dr. CARO, Dr. GNUECHTEL, Dr. LEHMANN []. Their application was investigated by Dr. FUCHS. 50X1-HUM
- e. Inorganic Developments: Insofar as they were applicable to the synthesis and development of dyes, the regeneration of sulfuric acid and the preparation of thionyl chloride and sulfuryl chloride were investigated by Dr. WOLFF.
- f. Continuous Processes: Plans and apparatus for the continuous production of p-nitraniline and of 2-aminoanthraquinone were developed by Dr. KRATZ and Dipl. Ing. BRINKMANN.
- g. Miscellaneous: Dr. KELLER investigated the carboxylation reaction of phenol to yield salicylic acid, whereas Dr. SCHUSTER and Ing. RANK investigated the oxidation of naphthalene to phthalic anhydride and the decarboxylation of phthalic acid to benzoic acid. To a small extent, synthetic detergents like "Igepone" and "Igepale" as well as the dye-fixative dicyanamide-formaldehyde water-soluble resin "Solidogene" were investigated by Dr. BRODERSEN. Fur dyes were investigated by Dr. LEHMANN. Certain insecticides and fungicides marketed under the tradename "Eulan" by Bayer, Leverkusen, were under development by Dr. MAIER-BODE.

C O N F I D E N T I A L

C O N F I D E N T I A L

- 5 -

50X1-HUM

- h. Services: An analytical laboratory under the direction of Dr. A. RICHTER was at our disposal. His previous work in Germany was with the development of ion-exchange resins, but he did no work on them while at the NIOPiK Branch Laboratory. However, his special knowledge in dehydration techniques was often of great assistance. The physicist of the group, Dr. FUCHS, was occupied with various problems in instrumentation.

11. [] the NIOPiK Branch Laboratory [] 50X1-HUM

- a. Development of an Analytical Method for the Determination of meta-Nitrochlorobenzene in an Isomeric Mixture. The technical nitration of chlorobenzene yields only about two per cent of the meta isomer, and although the chlorination of nitrobenzene is preferred because it gives better yields of the meta isomer, [] the Chimkombinat plant (on the basis of the results of the analytical procedure [] developed) that a grade of meta-nitrochlorobenzene of purity sufficient for the preparation of the dye Sulfur Black could be obtained by modification of the existing process to include a regenerative step. It would have been best if the meta derivative could have been isolated by an efficient low-pressure distillation, but this equipment was not available in the plant. [] analytical method was based on the determination of the freezing points of a ternary mixture of isomers, previously freed of nitrobenzene and dinitrochlorobenzene by distillation. Attempts to enrich the meta content by selective hydrolysis with steam of the ortho and para isomers was not successful. [] analytical method was not previously described in the literature that was available []; the reports of the I. G. Farben and reports issued by the Chimkombinat. 50X1-HUM 50X1-HUM 50X1-HUM
- b. Isolation of meta-Nitrotoluene from Technical Mixtures. In this case it was not necessary [] to conduct laboratory work, because the reports from the I. G. Farbenindustrie that were available [] described an excellent procedure for the isolation of pure meta-nitrotoluene by distillation. [] number of recommendations to the plant to this effect, including an analytical procedure for the meta-isomer. There was available to the plant the use of a 53-plate bubble-cap column that had been delivered to Chimkombinat for reparations; and although this column was quite suitable for vacuum distillation and separation of the three isomers, the plant was unable to make it achieve the desired separation. [] 50X1-HUM
- [] Consequently, the plant never did succeed in preparing the pure meta isomer.

C O N F I D E N T I A L

C O N F I D E N T I A L

- 6 -

50X1-HUM

c. Development of a Process to Produce Ethyl Acetoacetate:

A suitable process was designed for the preparation of ethyl acetoacetate from ethyl acetate and anhydrous sodium ethylate, based on reports from Hoechst and from the NIOPIK-Moscow laboratory. The process was tested at Moscow, and pilot plant erected at an unknown location.

50X1-HUM

d. Development of a Process to produce Phthalodinitrile:

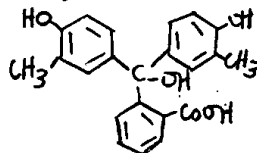
A suitable process was recommended, based on reports originating from Ludwigshafen and from NIOPIK-Moscow, and involved the catalytic dehydration of the reaction product of phthalic anhydride and ammonia. The original process called for bauxite, or activated alumina; but since this was not available in the USSR, a process based on phosphoric acid as a contact catalyst was recommended. This process also was tested at NIOPIK-Moscow,

50X1-HUM

e. Preparation of Indicators in a Laboratory Scale:

Industrial literature was not available, and details for the preparation of such indicators were obtained from the open literature.

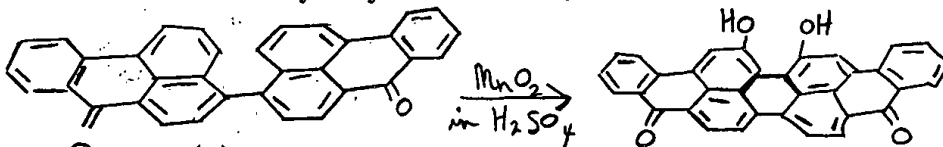
- (1) o-Cresylphthalein: Approximately 200 grams of this indicator were prepared by the condensation of phthalic anhydride with o-cresol, with toluene sulfonic acid as a catalyst.



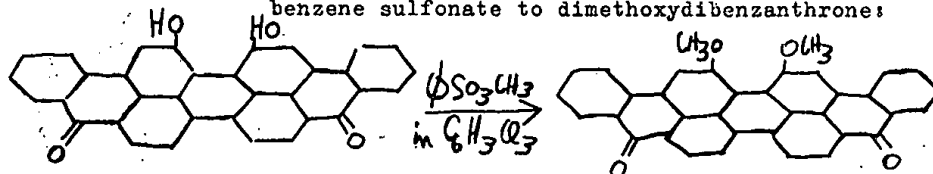
- (2) Bromoresol Green (Tetrabromo-m-cresolsulphophthalein): Attempts to prepare this common indicator from saccharin as a starting material were not successful.

f. Special Synthesis of Indanthrene Brilliant Green FFB: (Dimethoxydibenzanthrone). (Russian name: Kubovi-Yarko-Zelenyi C). The steps that were of interest to the plant were:

- (1) The oxidative condensation of dibenzanthronyl to dihydroxydibenzanthrone:



- (2) The methylation of dihydroxydibenzanthrone by methyl toluenesulfonate or in both cases (HO) benzene sulfonate to dimethoxydibenzanthrone:



C O N F I D E N T I A L

C O N F I D E N T I A L

- 7 -

50X1-HUM

This dye was produced in the plant, and the objective of this particular project was to clarify the reaction mechanism and to produce a standard sample of high-quality dye. A further objective was to suggest means of diminishing the consumption of sulfuric acid in step (1); and [] the sulfuric acid consumption could be minimized if the quality of the manganese dioxide used in step (1) were raised such that it was free of silica, with the consequence that the purification step be eliminated [] that required that the dihydroxydibenzanthrone be dissolved in concentrated sulfuric acid and reprecipitated by the addition of water, []

50X1-HUM

50X1-HUM

[] This research was guided by a plant report from I. G. Ludwigshafen, a short note from CIBA, and a prewar report from the NIOPiK, Moscow, laboratory. []

50X1-HUM

50X1-HUM

On the whole, the quality of the Indanthrene Brilliant Green FFB that was produced at the plant was not up to German standards. In an effort to correct this condition, [] the fact that the fastness of the dye would be affected by impurities introduced during manufacture; []

50X1-HUM

50X1-HUM

In general, the plant experienced a great many difficulties in manufacture, to judge from the fairly large number of questions []

50X1-HUM

6. Development of Methods for Manufacturing Anthrasole Dyes: These were vat dyes that were applied to the fiber in the form of the water-soluble hemisulfate of the leuco (reduced) form, and then oxidized in situ. This class of dye apparently was unknown in the USSR, [] the reports issued by I. G. Ludwigshafen in order to conduct the development work. The following dyes, neither of which were new, were developed [] for Soviet use []

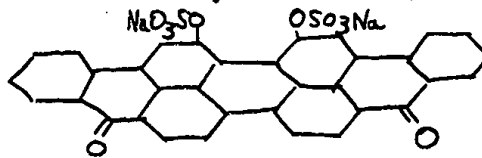
50X1-HUM

50X1-HUM

50X1-HUM

The dyes were:

- (1) Anthrasole Brilliant Green FFB (Russian name: Kybozom Yarko Zeleynyi C), or hemisulfate of leuco dimethoxydibenzanthrone:



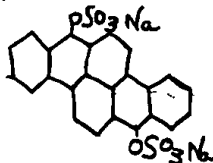
C O N F I D E N T I A L

C O N F I D E N T I A L

- 8 -

50X1-HUM

- (2) Anthrasole Golden Yellow GK (Russian name: Kybozom Zolotisto Zheltyi JK), or hemisulfate of dibenzpyrene quinone:



The hemisulfates were obtained by reduction of the parent vat dye with a metal powder in anhydrous pyridine, with simultaneous sulfation by means of chlorosulfonic acid. In connection with this research, [redacted] a procedure for the preparation of anhydrous pyridine by azeotropic distillation of the wet pyridine, and also developed preparative procedures for the preparation of dimethylaniline-p-sulfonic acid and of "Trioloxyd" (the polyethylene oxide derivative of triethanolamine).

50X1-HUM

12.

[redacted]

50X1-HUM

C O N F I D E N T I A L